

INVESTIGATION ON ADSORPTION OF NITRITE, NITRATE AND AMMONIUM IONS FROM AQUEOUS SOLUTIONS ON CARBON ADSORBENTS

ABSTRACT

The aim of the dissertation was to study the dynamic adsorption of nitrite, nitrate and ammonium ions from aqueous solutions on four modified carbon adsorbents. Commercial carbon adsorbent AG-5 (*GryfSkand*, Hajnówka) was used for the research. The research was carried out to determine the possibility of using these carbons for the separation and enrichment of nitrate ions from aqueous solutions before their further chromatographic analysis. The experiments were conducted for distilled and deionized water (after ion exchange), tap water, infusion liquid, and alcoholic beverages.

The theoretical part of the dissertation focuses on the selected issues related to the presence of nitrogen compounds in nature and their impact on the human. There are also cited the most important directives, in the context of the European ones, which aim at improving water quality, particularly drinking water, in terms of nitrate content. The presence of nitrite and nitrate ions in water and food is discussed, as well as the methods for their determination.

Additionally, the theoretical part of the dissertation describes the adsorption of ions on activated carbons from aqueous solutions. Also, carbon adsorbents has been characterized by presence of the oxygen functional groups on their surface, that are responsible for ion exchange capacity. The ion exchange process taking place in the dynamic system is also presented.

The experimental part of the dissertation describes the preparations of the carbon adsorbents obtained on the basis of carbon adsorbent AG-5. The adsorbent was crushed and sieved to obtain a fraction of $0.04 \div 0.063$ mm. Then, it was cleaned from surface contamination with an alcohol solution of concentrated hydrochloric acid in a Soxhlet apparatus, and suitably modified. It was oxidized with a 30% H_2O_2 solution (in boiling point

and in room temperature) and heated at 600°C under a nitrogen atmosphere. In this way, the surface was enriched with cation and anion exchange groups. The content of the cation exchange groups increased to 1.77 mmol·g⁻¹ (AG-5_2), and the anion exchange ones to 0.37 mmol·g⁻¹ (AG5_4).

For all the carbon adsorbents, the basic parameters of the porous structure were characterized on the basis of low-temperature nitrogen adsorption isotherms (ASAP). The percentages of carbon, hydrogen, nitrogen, and sulphur were determined by Elemental Analysis (CHNS). The concentration of the surface oxygen functional groups was determined by the Boehm method. Next, the equilibrium pH of the aqueous extract (pH_{PZC}) was marked. To confirm the presence of the functional groups on the surface of the analysed adsorbents, the Fourier-Transform Infrared Spectroscopy (FTIR) was performed. Additionally, the structure and surface morphology of the tested carbon adsorbents were examined using X-ray Diffraction (XRD) and Scanning Electron Microscopy (SEM).

The next part of the dissertation presents the results of the dynamic adsorption studies of nitrite, nitrate and ammonium ions on SPE columns packed with carbon adsorbent series AG-5. On the basis of adsorption isoplanes, the sorption capacities of individual carbons were calculated.

The adsorption of nitrate and ammonium ions on the other adsorbents was also investigated. As a tested adsorbents: active carbon (obtained from date palm leaves *ACDL*), carbon nanotubes, florisil, halloysite and porous copolymers with various functional groups were used.

The influence of selected parameters on the adsorption of nitrate and ammonium ions on carbon adsorbent series AG-5 was also investigated. The parameters included in the study were: ion concentration, adsorbent particle size, porous carbon structure and the content of surface functional groups.

An important aspect of the experiments was to investigate the suitability of the carbon adsorbent AG-5_1 for the isolation and enrichment of nitrate ions from aqueous solutions of the concentration below 1 mg·L⁻¹ using Solid Phase Extraction (SPE). Selected matrices were tested: distilled water, deionized (in the ion exchange process), alcoholic products (vodka, wine, tinctures) and waters with higher contents of other ions, i.e. infusion liquid, tap water. Based on the obtained test results, the degree of enrichment and recovery of nitrate ions from the tested matrices was determined.

This investigations shows that the adsorbent AG-5_1, with the highest sorption capacity in relation to nitrate ions, can be used to isolate and enrich nitrates from matrices with a low

content of other ions by solid phase extraction. The degree of enrichment was obtained within the limits of 260-470 times, for the selected matrices tested.

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